

TRANSMISSION ELECTRON MICROSCOPIC STUDIES OF  
SHAPE MEMORY STRUCTURES IN NiTi ALLOYS

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## Monterey, California



# THESIS

TRANSMISSION ELECTRON MICROSCOPIC STUDIES  
OF SHAPE MEMORY STRUCTURES  
IN NiTi ALLOYS

by

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March 1975

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Transmission Electron Microscopic Studies  
of Shape Memory Structures  
in NiTi Alloys

by

Robert Ripley Allen  
Lieutenant, United States Navy  
B.S., University of Idaho, 1967

Submitted in partial fulfillment of the  
requirements for the degree of

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## ABSTRACT

The reverted microstructure of equiatomic NiTi is characterized for several thermal and thermomechanical situations. The generation of dislocations is shown to occur in the martensite as well as the matrix. The debris left after reversion indicates several structures act together to bring about the conditions required for SME. The nature of this interaction is affected by prior thermal history.



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## I. INTRODUCTION

Some materials that undergo martensitic transformations have been observed to recover their original shape after being deformed. This effect is known in general as the "Shape Memory Effect", and is defined as the strain behavior of a material deformed beyond an apparent "yield point", that recovers most, if not all, strain upon stress release, and/or on heating [1]. Different investigators have observed a number of separate effects which fit the general description. This has caused some confusion in nomenclature. The different effects and their descriptive titles are presented in several recent reviews [2 - 6]. Perkins et al [1], to avoid lay terms for SME, suggested the terminology "thermoelastic martensitic reversion" or "martensitic thermoelasticity" as more scientific terminology. This terminology will be adopted in this paper as (TMR) and (MT), respectively, along with the general term SME. For the purpose of this paper, the "Shape Memory Effect" (SME) will be defined as the strain reversion requiring heating to some temperature above the deformation temperature. This definition does not preclude other effects being present only that reversion must be present upon heating above the deformation temperature.

Since 1951, the SME has been discovered in several alloy systems which include CuZn [7], InTi [8], FePt [7], AgZn [9],



AgCd [10, 11], and NiTi based "Nitinols" [12]. The near equiatomic NiTi system has been studied extensively since the first observation in 1961 by Buehler and Wiley of the Naval Ordnance Laboratory [12]. This system because of its good engineering properties is the subject of the present investigation.

The structure of near-equiatomic NiTi intermetallic compound has been established by most investigators as the CsCl, B2, structure. There are, however, still many questions to be answered about the martensitic structure. In 1961 Purdy and Parr reported a low-temperature hexagonal martensite [13]. Then Dautovich and Purdy [14] reported a triclinic structure. Later Marcinkowski, et al reported a structure occurring by simple shear on (112) and in 111 directions of the parent B2 structure, thus converting the original lattice into two distinct monoclinic martensites [15]. Wang, et al confirmed the existence of two structures and determined them to be triclinic and hexagonal [16]. Nagasawa reported the existence of a high density of stacking faults, which suggested a layered structure with stacking sequences of 4H, 2H, 3R, 12R and 18R showing hexagonal and rhombohedral symmetry [17]. Otsuka, et al reported a monoclinic unit cell [18]. The recent work of Zijlstra, et al [19] added a new martensite which they described as "Wavy" martensite, and suggested that the existence of at least three different types of martensite in TiNi might



account for the conflicting results reported by different investigators.

The above brief summary depicts a rather complex structure for the martensite phase, which in the macroscopic view presents some interesting effects. One of these effects is the increase in flow stress after a martensitic transformation, or after prestraining while in the martensitic phase. Rozner and Buehler found improved room temperature strength after prestraining NiTi at temperatures in the liquid nitrogen range [20]. A similar study by Ball, et al found prestraining at room temperature also improved high temperature strength [21]. This same study also noted that thermal cycling to liquid nitrogen temperature without prestraining increased the high temperature strength. These two studies indicate a generation of dislocations during the martensitic transformation, both for the thermal and strain induced transformations. The present transmission electron microscopy study was conducted to characterize the substructure of the reverted matrix, after thermal and strain induced martensitic transformations, at both liquid nitrogen and room temperatures.





## II. EXPERIMENTAL PROCEDURES

The alloys in this study were provided by Raychem Corporation, Menlo Park, California. The alloy rods were prepared by beam melting of a 1.25-inch diameter ingot, hot swaging to 0.5 inches, cold drawn in 15% passes to 0.0968 inches with intermediate anneals. The wire rods were then vacuum annealed at 900 C for thirty minutes. Alloy compositions were alloy "A" 49.2 Ti, 49.8 Ni, and 1.0 Al, and alloy "B" 49.5 Ti, 49.5 Ni, and 1.0 Fe (all atomic percent), with  $M_s$  of 0 C and -70 C respectively.

Bulk samples were thermal cycled by immersion in free boiling liquid nitrogen, and oil bath heated to 200 C. Thin samples were cycled by immersion in free boiling liquid nitrogen and heated to 200 C by heat gun. The heat gun setting and temperature vs distance was calibrated by mercury thermometer with a repeatable temperature of  $210 \text{ C} \pm 10 \text{ C}$ .

Mechanical testing was performed in tension on an Instron testing machine. The test samples were in rod form cut to two inches with a 1-inch gage length between grips. The grips used were of the collet type and were checked for slippage on each run. The strain was measured by using a Bentley-Nevada proximeter. Further details concerning thermal environment control, strain measurement, data, recording, calibration, and characteristics of the apparatus can be obtained from Reference 22.



Thin sections were prepared by first spark, cutting on an S. M. D. Servomet Spark machine to  $0.045 \pm 0.005$  inches, then thinned using a Buehler Electromet thinner with jet polishing. A 92% acetic, 8% perchloric acid solution at 12 volts was used [17]. The thinned sections were washed in distilled water and dehydrated alcohol. The possible structural disturbance of the spark cutting was checked by examination of the transmission microstructure of the as-received rods. No detectable effect was noted [Figs. 3 and 4].

All foils were examined at room temperature using a tilting stage in a JEM-7 Japan Electron Optics Laboratory electron microscope operating at 100 kv.



### III. RESULTS

The annealed rods of alloy "A" and "B" had featureless microstructures with no dislocation arrays or martensite plates observed. The room-temperature structure was the CsCl "B2" structure [Figs. 3 and 4].

Thermal cycling of alloy "B" from 22 C to -196 C to 22 C (fully below  $M_f$  to fully above  $A_f$ ) generated dislocations aligned on at least two slip plane variants [Figs. 5 and 6]. When the number of full thermal cycles was increased, the dislocation network developed further and retained martensite plates were observed in alloy "B" [Figs. 7 and 8].

Thermal cycling of alloy "A" over the same temperature range did not develop the same microstructure as alloy "B". This was expected in that these cycles were "partial" (i.e., fully below  $M_f$  but not to fully above  $A_f$ ). After one partial cycle (22 C to -196 C to 22 C) the reverted structure contained some thin parallel-sided martensite plates [Fig. 9], but no dislocation arrays. Further partial cycling revealed plates of the twinned or layer type martensite [Fig. 10]. The same sample was then cycled from 22 C to 200 C to 22 C. After one cycle, dislocation networks were observed throughout the thin section [Fig. 11]. Multiple full cycling from 22 C to -196 C to 200 C to 22 C generated a structure similar to multiple full-cycled alloy "B" (cycled 22 C to -196 C to 22 C). For illustrations of this point, compare Figures 5 and 6 with 13, 14 and 15.



The reverted structure of alloy "B" after straining varied with the percentage strain; at low strains (2% or below) the structure was similar to the thermally cycled structure [Fig. 16]. When the strain was increased onto the plateau region of the stress-strain curve (thermoelastic martensite region), the dislocation network appeared to be closely aligned with the reverted martensite plates [Fig. 17]. Straining beyond the plateau region caused the ordered dislocations to form random high density dislocation tangles [Figs. 18 and 19]. Figure 20 shows dislocations piled up at grain boundaries due to plastic flow.





#### IV. DISCUSSION

The martensite transformation of near equiatomic TiNi has been well established by many previous studies [13, 14, 15, and many others]. These same studies have found various types of martensite structures, many of which are similar, but with different growth and internal characteristics. The transformation from the CsCl "B2" structure to the martensite structure would, all things being equal, increase the volume of the total matrix. This increase in volume would develop high stresses in both the surrounding matrix and the growing martensite plate, either of which could at some point exceed its yield point, with the appropriate structural adjustment. The present study and others [21, 23] have found dislocations in the reverted structure after both athermal and deformation martensite transformations. The present study would indicate that dislocations are generated both internal to martensite plates and at martensite-matrix interfaces, depending on the circumstances. This generation of dislocation debris would strengthen (work-harden) the matrix, an effect which has been observed by Ball, et al [21], the present study, and by Cross, et al [24]. Cross noted a decrease in the percentage shape recovery ( $\%R = (\epsilon_i / \epsilon_R) \times 100$ ) after both thermal and deformation cycling, with the largest decrease ( $\epsilon_i - \epsilon_R$ ) on the initial cycle, and diminishing with subsequent cycles, until after  $n$  cycles ( $\epsilon_i - \epsilon_R$ ) $_n = 0$ ,



thereafter maintaining "perfect" TMR. The reason for this effect, it now appears, is the generation of dislocations by transformation cycling, with a stable substructure of tangled dislocations becoming established after a given number of cycles. The result is a true thermoelastic behavior, which depends on the deformation amplitude.

It is postulated that several martensitic structures acting together account for the debris left after reversion and ultimately the TMR. Just how the various structures relate one to another, and why one is more-or-less prominent in a given alloy, thermal cycle, or deformation process requires a more intensive study. However, some understanding can be gained by relating various reported structures to the reverted structure found in this study. The structures required are found in the following reports: Otsuka, et al [18] found internally twinned martensite, and also noted martensitic structures with stacking faults. Marcinkowski, et al [15] reported a martensitic structure generated by equal but opposite shears in the  $[\bar{1}11]_{B2}$  and  $[1\bar{1}\bar{1}]_{B2}$  directions, on the  $(1\bar{1}2)$  habit plane. The important features to note (as pointed out in the report) are the complete absence of dislocations, faults, or twins with this structure. Gupta, et al [25] found a microstructure of two monoclinic martensitic structures in the form of alternating platelets with dislocations generated at the interface between the alternating bands. This same study also reported the



presence of at least six different martensitic structures in equiatomic TiNi alloys. The results of the present study can now be discussed using the above structures as the basis for the debris found in the reverted structure.

The reverted structure of thermally cycled alloy "B" had areas which contained debris left by twinned martensite, and other areas that were free of debris. Figure 5 shows two debris areas separated by a debris-free band which must have contained some other structure. The debris-free band could be an area where the Marcinkowski structure had been. In Figures 7 and 8, some retained martensite plates are present, which appear to be the layered structure of stacking faults found by Nagasawa [17]. Alloy "B" did not show much debris from the banded martensitic structure when thermally cycled.

Alloy "A" had a high percentage of the banded (Gupta) related debris [Figs. 11, 12, and 13]. This was especially true for the stepped thermally cycled samples [Figs. 11 and 12]. The samples of the fully thermally cycled rods had debris of all three structures [Figs. 13, 14, and 15].

The absence of twinned structural debris in the partially cycled samples of alloy "A" was not expected, but did provide some support for the close association of dislocation generation and the martensite plates. Ball, as mentioned previously, had shown that thermal cycling of NiTi increased the elastic flow stress. The above observations and known results lead to the following predictions:





1. The flow stress would be higher for the fully thermally cycled material.

2. The partially cycled material would have a higher flow stress than the as-annealed rods, but not as high as the fully cycled rods.

3. The partially cycled rods would have, because of incomplete transformation, increasing irregularities in the plateau region of the stress-strain curve caused by some type of yet unknown transformation.

Figure 1 shows the results of the tension tests conducted at room temperature. The results were exactly what was predicted. These tests also showed a decrease in the stress at the end of the plateau for the thermally cycled samples (for fully cycled the drop was less than for the partially cycled), but not in the as-annealed sample. The work-hardening rates after the plateau are also different; no explanation for this phenomenon will be offered at this time.

The microstructure of the reverted strained alloy "B" samples varied depending on the percentage strain. At two percent strain the structure was much like the thermally cycled reverted structure [Fig. 16]. Increasing the strain to the plateau region left a reverted microstructure that indicated an interaction of different martensitic structures [Fig. 17], the exact nature of which is not understood. It is believed that the wedge shape is the edge of a reverted twinned martensite structure, which is growing under the



action of the applied stress at the expense of the banded monoclinic structure. The resulting interaction has left a high density of dislocations along the wedge front. This process is continued until all the matrix is transformed. Straining beyond the plateau detwines the structure. Figures 18 and 19 show the reverted matrix containing only the twinned debris. At ten percent strain, some plastic flow has occurred as evidenced by Figure 20.



## V. SUMMARY AND CONCLUSIONS

A transmission electron microscopy investigation has been made of the reverted martensite structure occurring in the equiatomic NiTi alloy. The debris left after reversion indicates several structures act together to bring about the conditions required for TMR. The generation of dislocations occurs during thermal and deformation transformations, and dislocations are generated in both the matrix and in the martensite. The plateau region of the stress-strain curve is the result of several martensitic structures interacting (under the application of an applied stress) with the nature of this interaction affected by prior thermal history. The overall transformation could be described as a "transelastic stabilization interaction" (TSI) which could account for various observed mechanical behaviors as the strain-, temperature-, and time-dependence of parameters such as reversion stress and strain.

The reversion stress for a given alloy was found by Cross, et al [24] and Johnson [26] to be a function of both temperature and percentage strain. The reversion stress was found to increase with increased initial strain up to some maximum strain level, beyond which the reversion stress decreased. This type of behavior is consistent with the reversion structure observed in the present study. The reversion stress from the TSI standpoint would be a function of the twinned martensite present, and the type of dislocation



network developed (both of these phenomena are functions of temperature). Thus, the reversion stress would increase as the percentage of twinned martensite increased, and would require a higher temperature to reach this stress level because of the dislocation development which would stabilize the thermoelastic plates. The maximum reversion stress would be reached when the transformation to twinned martensite is complete and before the development of the highly tangled dislocation networks. When the tangled dislocation networks start to develop (% strain depends on alloy), the reversion stress (internal stress) is effectively relieved, i.e., there is a stabilizing effect of the dislocation arrays at the matrix interface. The possibility of stabilized thermoelastic martensite remaining in the matrix above  $A_f$  and below  $M_d$  would decrease the reversion stress at a given temperature in this range. Cycling the material either thermally or by deformation (below plastic flow) stabilizes the transformation giving the hysteresis curves found by Cross. Johnson did not cycle the material and did not get the well-formed hysteresis curves that Cross did, which indicates that cycling does indeed stabilize the transformation. The type of cycling (thermal or deformation) which would give the response desired requires more mechanical-microstructure correlation.

The stabilizing affect of cycling (both thermally and deformation) on the strain recovery as found by Cross





(mentioned previously) is also consistent with the results of the present study. The magnitude of the decrease in percentage strain recovery (i.e., the extent of imperfect thermoelasticity) decreases with increasing cycles, due to increasing work hardening of the matrix. When after  $n$  cycles, the matrix yield stress becomes equal to or greater than the internally developed transformation stress, no further plastic flow occurs, and a constant percentage strain recovery (perfect thermoelasticity) is observed.

The correlation of the martensitic structures present in the plateau region with the reverted debris, as a function of strain, would aid in the determination of the process which causes the thermoelastic behavior of equiatomic NiTi alloys. This also would be the recommended first step in determining the cycling-to-mechanical-behavior relationship, thus allowing a better prediction of mechanical properties for a given alloy, and also facilitating the type of thermal cycling and forming processes required for the increasingly complex engineering problems that this material could solve.



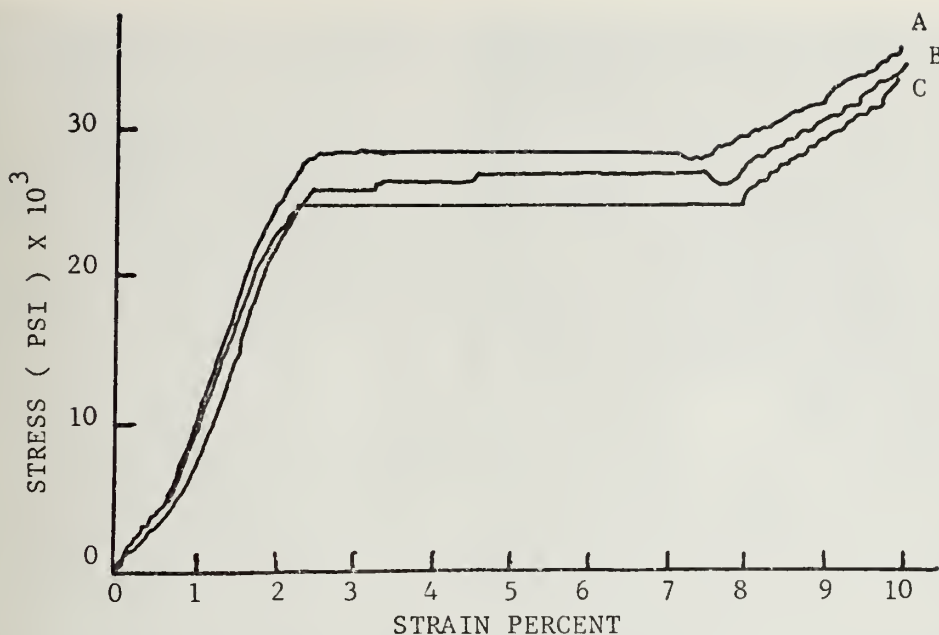


Figure 1. Alloy "A" tension test, room temperature: (A) Full thermal cycle; (B) Partial thermal cycle; (C) Annealed.

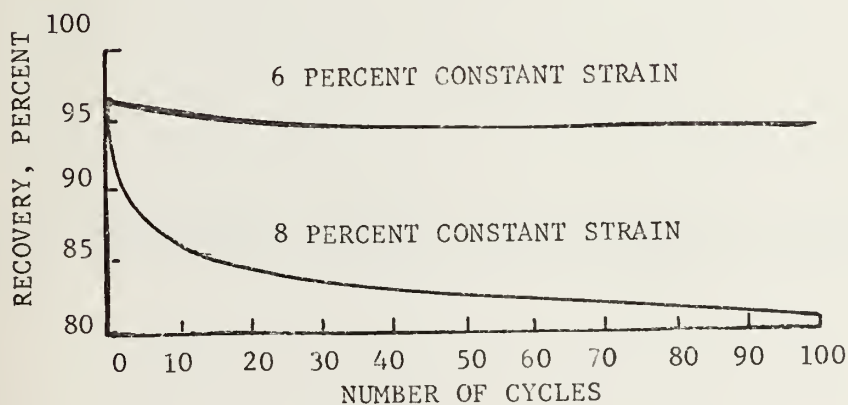
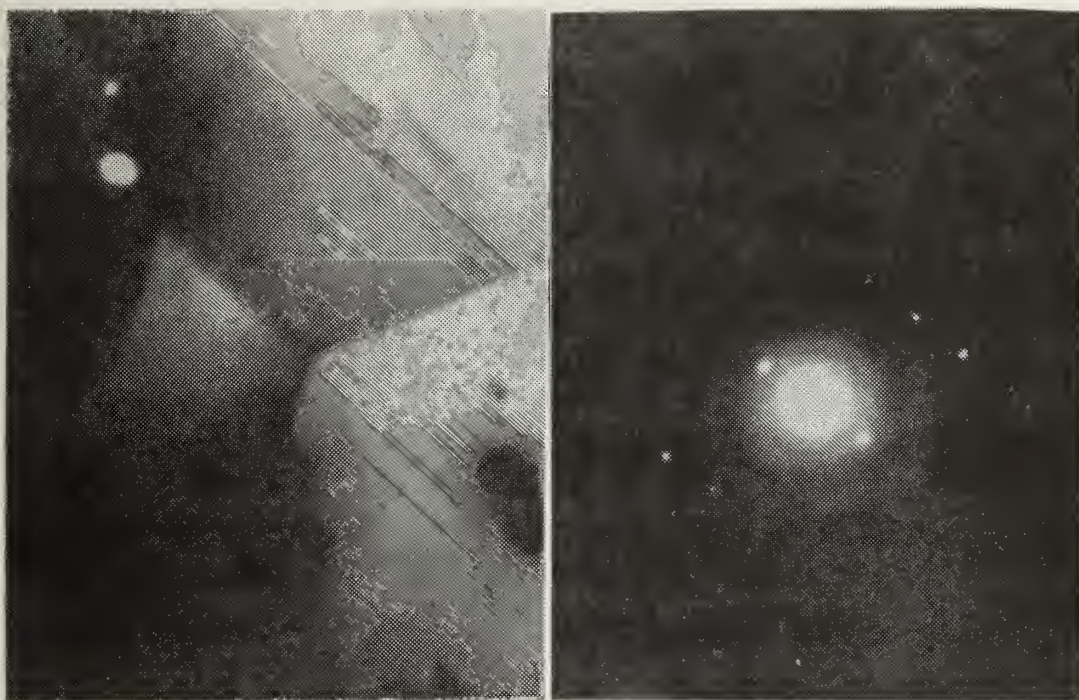


Figure 2. Tensile shape recovery of 0.020 inch wire after successive strain-heat-cool cycles [Ref. 24].





(A)

(B)

Figure 3. Alloy "B" annealed: (A) BF image 25,000X; (B) SAD.







(A)



(B)

Figure 4. Alloy "A" annealed: (A) BF image 15,000X;  
(B) SAD.





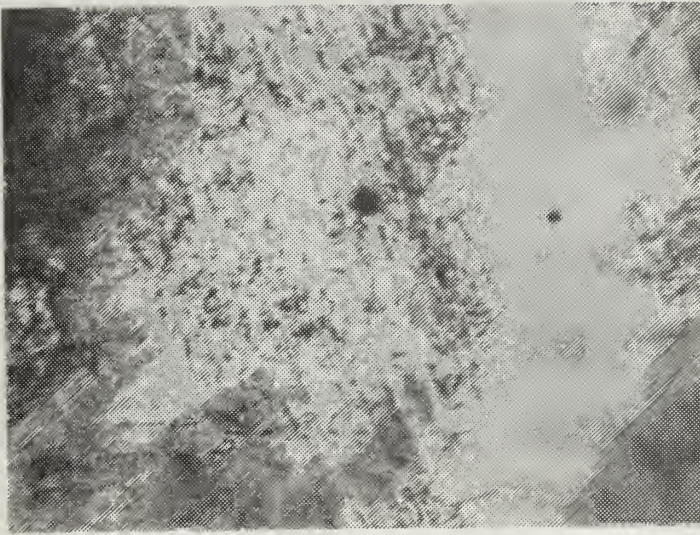


Figure 5. Alloy "B" thermally cycled 22 C to -196 C to 22 C three times; BF image 35,000X.

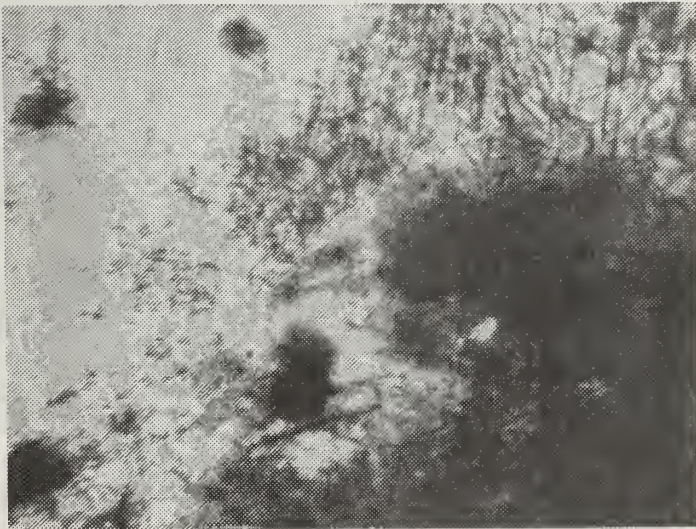


Figure 6. Alloy "B" thermally cycled 22 C to -196 C to 22 C five times; BF image 22,000X.





Figure 7. Alloy "B" thermally cycled 22 C to -196 C to 22 C five times; BF image 25,000X.



Figure 8. Alloy "B" thermally cycled 22 C to -196 C to 22 C five times; BF image 70,000X.







Figure 9. Alloy "A" thermally cycled 22 C to -196 C to 22 C once; BF image 7,200X.



Figure 10. Alloy "A" thermally cycled 22 C to -196 C to 22 C five times; BF image 12,000X.



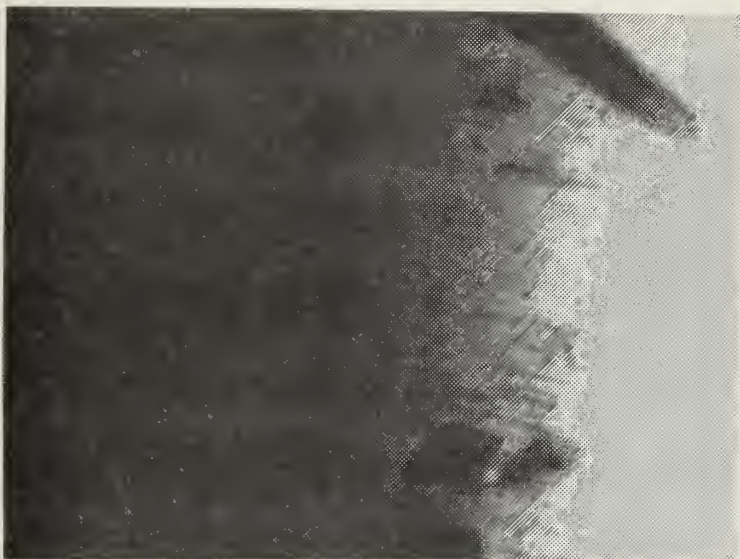


Figure 11. Alloy "A" thermally cycled 22 C to -196 C to 22 C five times, and 22 C to 200 C once; BF image 25,000X.

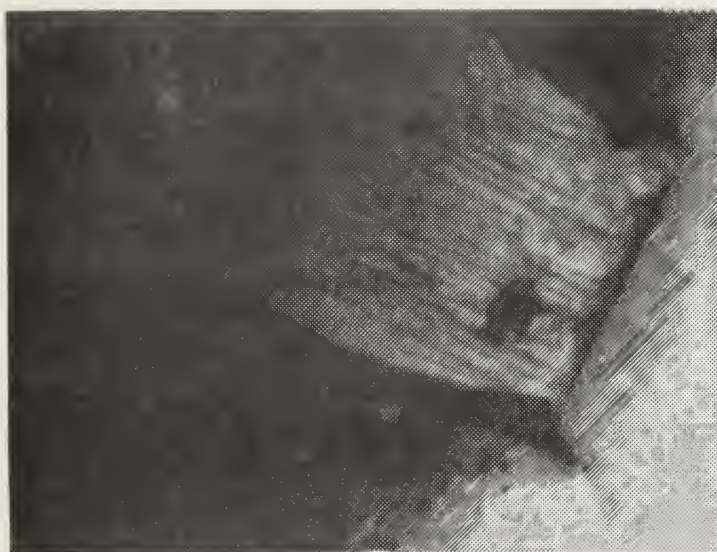


Figure 12. Alloy "A" thermally cycled 22 C to -196 C to 22 C five times, and 22 C to 200 C to 22 C once, BF image 25,000X.





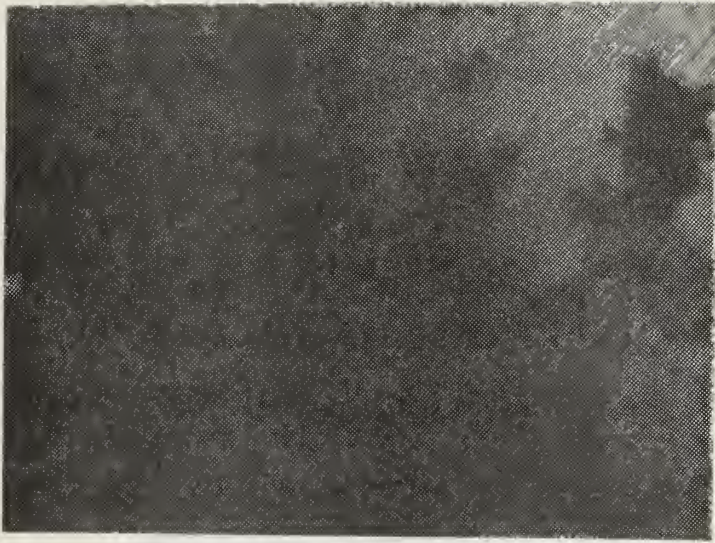


Figure 13. Alloy "A" thermally cycled 22 C to -196 C to 200 C to 22 C five times; BF image 42,000X.

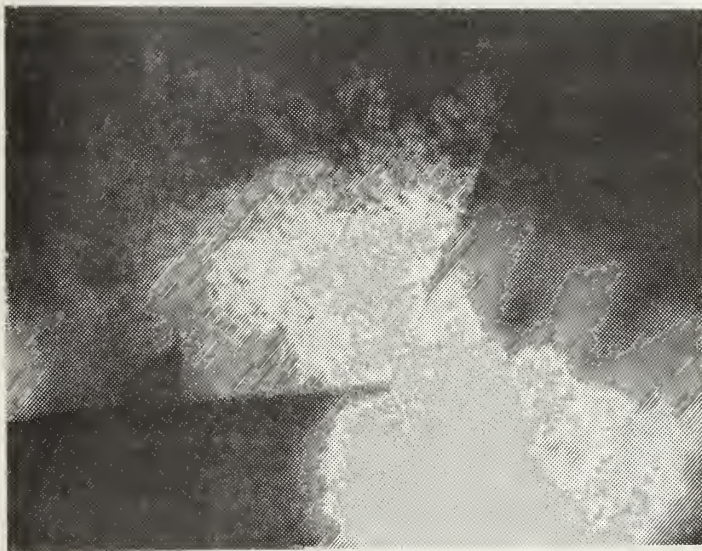


Figure 14. Alloy "A" thermally cycled 22 C to -196 C to 200 C to 22 C five times; BF image 57,000X.





Figure 15. Alloy "A" thermally cycled 22 C to -196 C to 200 C to 22 C five times; BF image 29,000X.





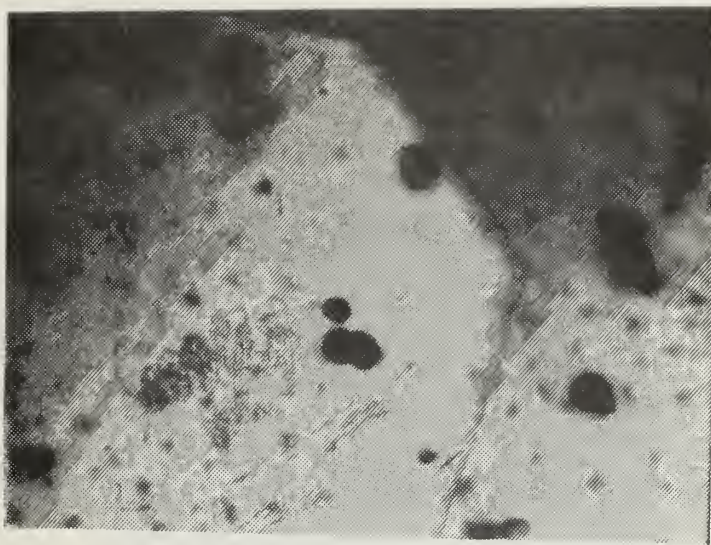


Figure 16. Alloy "B" strained to 2% at -196 C; BF image 13,700X.

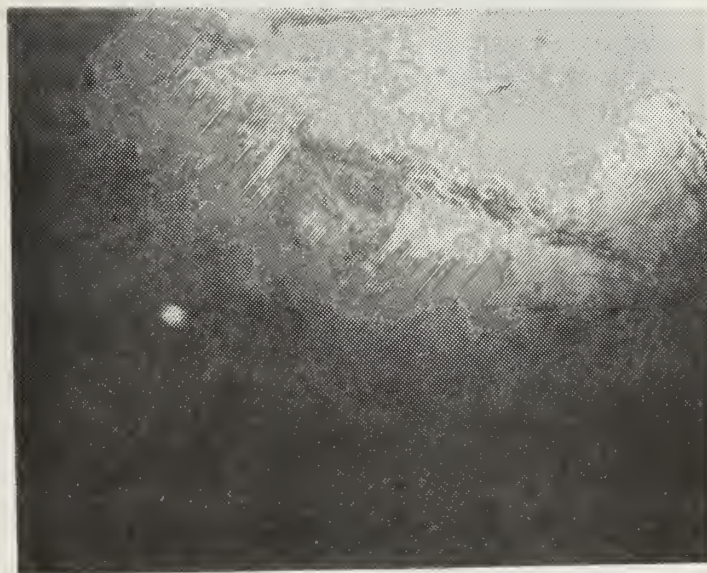


Figure 17. Alloy "B" strained to 4% at -196 C; BF image 8,500X.





Figure 18. Alloy "B" strained to 8% at -196 C; BF image 55,000X.





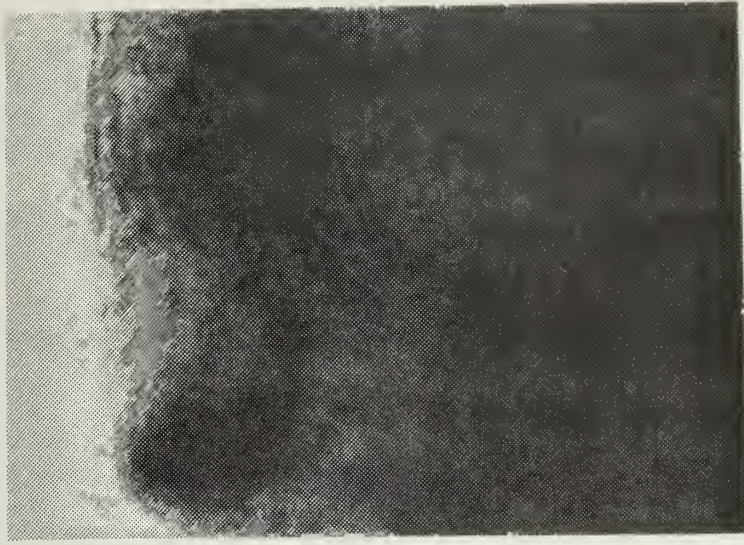


Figure 19. Alloy "B" strained to 10% at -196 C; BF image 22,000X.

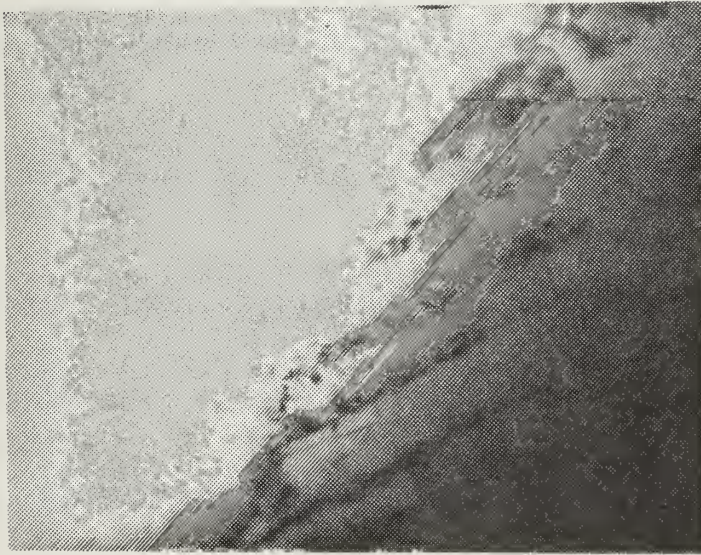


Figure 20. Alloy "B" strained to 10% at -196 C; BF image 39,000X.



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